

Gypsum-bonded Investment and Dental Precision Casting (Ⅲ) Composition of Investment for the Quick Casting Technique

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A simultaneous differential thermal analysis and thermogravimetry method previously established was used to estimate the composition of gypsum-bonded investment marketed for the quick casting technique. Three commercial investments of this type were heated to 700°C at 10°C/min and the hemihydrate content was estimated by the mass decrease reached at 300°C after subtracting the mass decrease at 100°C as moisture content. The hemihydrate contents were between 25% and 30%, which appears to be the range also chosen for the conventional gypsum-bonded investment of cristobalite type over 70 years by the industry. However, the new type of investment contained both cristobalite and quartz. The small sample size is a disadvantage of the present method but this can be overcome by more frequent use of the method by investigators.

Key words: Investment composition, rapid heating investment, DTA-TG

INTRODUCTION

It is not common to make reference to the composition of casting investment in our research. A simple method, which utilized simultaneous differential thermal analysis and thermogravimetry (DTA-TG), was reported to estimate the content of calcium sulfate hemihydrate incorporated in gypsum-bonded casting investment^{1,2)}. The method found that the content of calcium sulfate hemihydrate was about 30% by weight in two conventional gypsum-bonded cristobalite investments, the oldest and more recent, giving an estimation for the refractory content of about 70% by weight²⁾. In the present study, the above method was applied to find the composition of investment recently marketed for the quick casting technique (hereafter, rapid heating investment).

MATERIALS AND METHODS

Table 1 shows three rapid heating investments analyzed in the present study. Each investment sample and α -alumina reference material were kept in a desiccator over silica gel for at least 24 hr before the DTA-TG measurement (Thermoflex, Rigaku Co., Tokyo, Japan). A quantity of 200±0.1 mg was used for both sample and reference in each analysis. They were placed in two separate compartments of a platinum sample holder. Each compartment had a recess at the base to accommodate the hot junction of a thermocouple. The samples were heated to 700°C at a rate of 10°C/min. The TG display was 20 mg full

scale with an accuracy of 0.2%. Mass changes were calculated at every 100°C as percentage change. Drying over silica gel does not completely remove the moisture adsorbed by calcium sulfate hemihydrate (hereafter, hemihydrate), especially in the case of dry-calcined β -hemihydrate^{1,2)}. Based on this finding, the estimation of hemihydrate content was made from the mass decrease at 300°C after subtracting the mass decrease at 100°C as moisture content and then by comparing the result with the theoretical dehydration value of 6.25% of hemihydrate to anhydrite. As secondary information, peak temperature was also determined for each endothermic or exothermic reaction on the DTA curve with a display range of ±25 μ V. The temperature display was 1000°C full scale with an accuracy of 0.3%. At least 5 samples were tested for each investment. Ambient laboratory conditions during the analysis were 21±1°C and 50±10% relative humidity.

Table 1 Three rapid heating gypsum-bonded investments studied

Code	Name	Recommended w/p ratio
R1 ¹	Cristoquick II	0.33
R2 ²	Cristobalite PF	0.35
R3 ³	Cristobalite FF	0.35

¹GC Corp. Tokyo, Japan

²Shofu Inc., Kyoto, Japan

³Noritake Co. Ltd., Nagoya, Japan

Table 2 DTA-TG results of three rapid heating investments

Material	R1 (n=6)	R2 (n=5)	R3 (n=5)
Mass decreases (%)			
100°C	0.04 (0.04)	0.00 (0.00)	0.19 (0.01)
200°C	1.59 (0.07)	1.85 (0.04)	1.95 (0.02)
300°C	1.60 (0.07)	1.85 (0.04)	1.96 (0.02)
400°C	1.60 (0.07)	1.86 (0.04)	1.96 (0.03)
500°C	1.60 (0.07)	1.86 (0.05)	1.96 (0.03)
600°C	1.60 (0.08)	1.86 (0.04)	1.96 (0.02)
700°C	1.60 (0.07)	1.86 (0.05)	1.96 (0.02)
Peak temperature (°C)			
1st endotherm	184 (0.8)	184 (0.8)	182 (1.6)
2nd endotherm	255 (0.4)	250 (0.7)	233 (1.5)
3rd endotherm	570 (0.7)	569 (0.0)	570 (0.7)
Exotherm	198 (1.4)	200 (0.5)	198 (1.2)
Content of calcium sulfate hemihydrate (%)	25.0	29.6	28.3

Values shown are average (standard deviation).

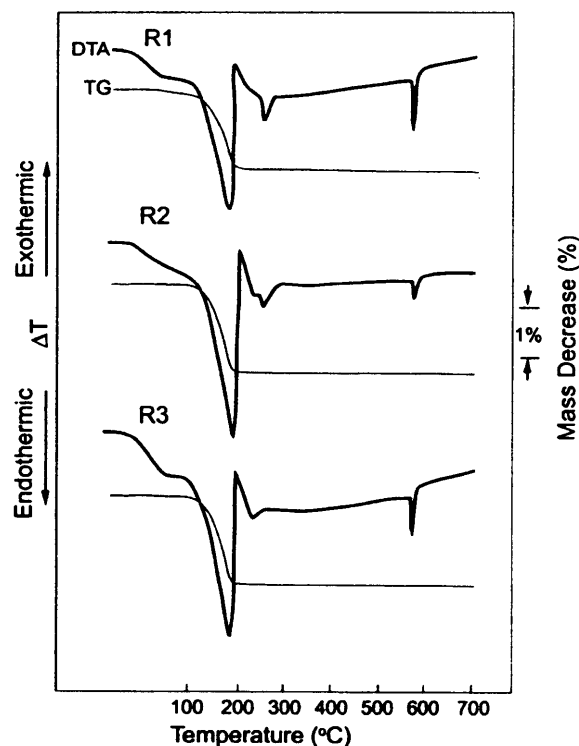


Fig. 1 Representative DTA-TG curves of three rapid heating investments. R1=Cristoquick II (GC), R2=Cristobalite PF (Shofu), R3=Cristobalite FF (Noritake).

RESULTS

Figure 1 shows representative DTA-TG curves of the three investments. Both DTA and TG curves are very similar for all materials. A large mass decrease, which was almost complete by 200°C, corresponded with a large endothermic reaction. This was the only significant mass change, apparently due to the dehydration of hemihydrate. Table 2 summarizes

results of the analysis. The mass decrease at 100°C ranged from nil (R2) to 0.19% (R3). This produced a small deflection on the DTA curve, most prominent with R3. The average peak temperature of the first endotherm due to the dehydration of hemihydrate was almost same, 182°C or 184°C, and an exothermic peak having an average peak temperature of 198°C or 200°C immediately followed the endotherm. Little mass decrease occurred after 200°C, giving a constant at 300-700°C in each material. In this temperature range, two endothermic reactions appeared at about 250°C and 570°C, indicating the α - β (low-high) inversion of cristobalite and quartz, respectively. The contents of hemihydrate calculated by the method described before were 25% (R1), 30% (R2) and 28% (R3) by weight.

DISCUSSION

The most extensive compositional analysis of gypsum-bonded investment was made on 38 commercial products in 1930³. Their thermal expansion (TE) values were less than 1% due to the high β -hemihydrate (plaster) contents, between 30% and 50% with many products, and also due to the use of quartz. This survey also showed the possibility of achieving a higher TE value of 1.25% with an experimental investment by replacing quartz with cristobalite. Three years later, one of the authors further reported the replacement of β -hemihydrate (25%) with α -hemihydrate (dental stone) to achieve a TE value of 1.5%⁴. Since that time, the content of α -hemihydrate seems to have remained in the range of about 25 to 30%, the latter value being the estimation by the present method for the two investments of cristobalite type as mentioned before². The same range of hemihydrate inclusion was found on the three rapid heating investments analyzed in the present study. However, the refractory component, 75%

(R1), 70% (R2) or 72% (R3), was a mixture of cristobalite and quartz in all materials, which is somewhat difficult to expect from their proprietary names. The inclusion of quartz was evident in the TE measurement of R1 and R2 under both general and rapid heating conditions⁵⁾.

The previous analysis²⁾ showed the possibility of using both α -hemihydrate and β -hemihydrate in the oldest commercial gypsum-bonded investment of cristobalite type, which dates back as far as 1934 to a report by Van Horn⁶⁾. The relatively large mass decrease of 0.19% at 100°C in R3, even after drying over silica gel, leaves this possibility of β -hemihydrate inclusion, although in this case the exothermic peak should appear at around 350°C under the same analytical conditions¹⁾. The inclusion, however, could be insignificantly low, as the recommended water/powder ratio of R3 is not particularly higher than those of the other two investments. Furthermore, even with significant inclusion of β -hemihydrate, its sluggish transformation from III-CaSO₄ to II-CaSO₄ could result in an unnoticeable size of exotherm. While the exotherm due to this transformation in α -hemihydrate was clearly shown at about 200°C (Fig. 1) due to its more rapid reaction than in β -hemihydrate, it is an impossible task to determine the ratio of the two types of hemihydrate existing in gypsum-bonded investment by DTA.

The primary aim of the present study was to apply the DTA-TG method previously established to estimate the composition of three rapid heating investments. The use of the mass decrease at 300°C was proved to be appropriate again, as there were no mass changes above this temperature. The analysis,

however, also provided a fresh insight into the role of gypsum binder in this casting technique; despite the higher hemihydrate content (30%) in R2 in comparison with that in R1 (25%), the former gave a higher average TE value of 1.93% against 1.72% in the latter⁵⁾. Accumulation of basic data will benefit us in further understanding of investment materials, as this example demonstrates.

One of the disadvantages of the present DTA-TG method is that the analysis is made on a small sample size. While this is a common DTA-TG technique, even distribution of investment components in the parent material must be assumed upon the adoption of data obtained. This disadvantage can be overcome by more frequent use of the present method by investigators.

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